Variability in composite materials properties

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Abstract. Composite materials are created as a quite complex architecture which includes a fibre reinforcement structure and matrix material. Many material parameters play a role when composite structures are modelled, e.g. in finite element models. In addition to the properties of the raw fibre and matrix materials which are used, also geometrical parameters have a significant effect on structural characteristics. Fibre reinforcement geometry together with material properties of fibre and matrix determine homogenised material properties.

The first part of the paper gives an overview of the most important processes which are used in composites processing industry. The factors which affect variability are also listed, and the effect of variability on material parameters is mentioned as well. The second part of the paper elaborates the identification of geometrical variability of the fibre reinforcement structure which is encountered with one particular type of composite material, namely a twill 2/2 carbon fibre weave with an epoxy matrix.

Introduction

Composite materials are available for several decades already. The many families of composite materials have just one thing in common: they have some kind of fibre reinforcement and some kind of matrix material to hold the fibre reinforcement in place. Apart of that, there are many different variants, which can be identified on three different aspects:

- fibre reinforcement: here again, there are different aspects
  - fibre material is a parameter, with glass and carbon as the most commonly used materials, but natural fibres such as flax and hemp have a very promising future
  - length of fibre: short or long
  - fibre reinforcement architecture: short fibre are usually organised in some random distribution, whereas long or continuous fibres are organised in a well-structured architecture
- materix material: there are two main categories
  - thermoplastic matrix, in which the polymer becomes mouldable above a specific temperature and solidifies again after cooling
  - thermoset matrix, in which a chemical reaction takes place
- processing technology: a wide range of technologies are available, with different characteristics

The three categories which are listed above are not independent. A particular processing technology is appropriate for one or several specific types of fibre reinforcement and for particular types of matrix materials. The particular choice of a fibre type and reinforcement architecture, matrix and processing technology depends on the application, with again a multitude of parameters which play a role: geometry of the component, specifications on structural performance, weight of the component, processing time, size of series, environmental requirements, recyclability, ... Obviously, cost of manufacturing and exploitation of the component is a very important factor.

For most modern products and applications, numerical analysis is done in the design phase to verify the performance of the product. In this paper, the particular case of structural characteristics is taken as a reference. Structural analysis is often done using finite element analysis. In order to be able to conduct a reliable design analysis, representative models are required.

The representation of a composite material requires many parameters, as listed above. Each of these parameters exhibits uncertainty and variability. This paper uses these terms as they are defined by Oberkampf et al. [1]. As many design parameters play a role, likewise many sources of non-determinism have to be taken into account, which implies that many model parameters have to be represented by a relevant non-deterministic model, either in a probabilistic format through a probability density function or in a non-probabilistic format through an interval number or a fuzzy number [2].

Probabilistic methods are used to describe scatter in properties. Probability distribution functions (pdf) can be established for all uncertain parameters, taking into account the correlation between different parameters. The result of the analysis can be interpreted in a statistical sense, and the probability of every output quantity depends on the input probabilities and their correlations. It is important that all these inputs must be validated in order for the result to allow for a statistical interpretation. Unfortunately this fact is often neglected by many scientists, and assumptions are made on the input pdfs [2]. The definition of an input pdf is then subjective, and so is the result. Freudenthal [3] states that "ignorance of the cause of variation does not make such variation random.". The availability of objective and validated data is thus required.

An additional factor is the distribution of a parameter within the structural component. Both structural parameters, such as plate thickness, and material parameters, such as Young's moduli, may not be constant within the entire structure. Physical reality usually implies some relation between a parameter value at a specific position inside the component and another position. In a context of probabilistic analysis, the concept of random fields is well suited to express spatial variability in a component [4]. In a context of non-probabilistic analysis, the concept of interval fields which is proposed by Verhaeghe et al. [5] offers an attractive perspective.

This paper starts with an overview of different processing technologies, all of which are well known. The focus of the discussion is on the sources of uncertainty and variability which are relevant for that technology.

The second part of the paper focusses on the quantification of variability in composite material stiffness properties. The methodology is applied on a typical woven textile composite.

Effects of uncertainty in composites production processes

The processing technique which is used in the manufacturing phase inevitably introduces uncertainty and variability in the mechanical characteristics of the composite product.

Composites technologies This section starts with an overview of the most important processes for composites manufacturing and processing. They are usually a combination of a fibre reinforcement architecture, a thermoplastic or thermoset matrix and a processing technology. Each of these processes has specific quality characteristics, which are discussed below. The term quality refers to the degree by which the product matches the pre-defined specifications. The present paper focusses on geometrical characteristics, and quality expresses how well product geometry matches the ideal geometry, especially when the process is executed repeatedly.

- low or high pressure injection moulding techniques, such as resin transfer moulding, vacuum infusion, RTM-light, injection moulding, reaction injection moulding all inject resin in a liquid state into a closed mould in which the fibre reinforcement is already present. Depending on the precise definition of the process, the fibre volume fraction of the composite may be variable.

The precise position and shape of the reinforcement is not well known because the resin which flows into the mould may shift or deform it, which implies that the exact orientation of the reinforcement is not guaranteed. An important aspect of this process is the development of the flow front, for which simulations are done to verify the quality of the process.

- high pressure compression moulding techniques, such as SMC (sheet moulding compound), BMC (bulk moulding compound) typically have a low fibre volume fraction. This is manufactured by dispersing long strands of chopped glass fibres or carbon fibres on a bath of resin (commonly polyester resin, vinylester resin or epoxy resin) before compressing them.

  The degree of variability is quite high, because the fibres are randomly oriented and the fibre volume fraction is variable within the volume of the component.

- material configuration techniques such as hand lamination, spray-up, prepreg autoclave curing, automated tape placement, filament winding, . . . all prepare the fibre reinforcement in a mould or on a mandrel.

  In manual lamination, the operator positions the reinforcement in the mould. The common type of reinforcement with hand lamination is a weave, with a structure which should be quite regular. However, the example in the next section of this paper demonstrates that individual tow paths do exhibit some irregularity, even within one weave. The manual procedure of positioning the layers in the mould introduces additional uncertainty, as the accuracy is limited. Also the thickness of the structure is not precisely known, especially when layers overlap each other.

  In automated tape laying, the same operation is done by a tape laying machine. The tape laying machine is either a gantry type robot or an articulated robot. The fibres are usually arranged in a uni-directional orientation, as they come from the tape which is unrolled from the tape layer. The quality of automated processes is obviously much better controlled than with manual processes. Adjacent tapes are well positioned next to each other, and the automated process guarantees that there are no overlaps. The orientation of the tape is also quite accurate, as it coincides with the moving tape layer head.

  In the filament winding process, each long fibre is placed individually on a rotating mandrel in a continuous process. Position control is thus quite accurate and also the orientation of the fibre is close to the nominal design. Thickness may exhibit some variation because the contact pressure between the fibre and the mandrel is not well controlled. And the amount of resin which is applied on the mould cannot be precisely controlled either.

- continuous production, with technologies such as pultrusion and continuous lamination: fibre reinforcement is applied along the longitudinal direction of the component.

  The fibre is mainly aligned with the production direction of the material, but the precise orientation is not exactly known.

- sandwich construction consists of minimum three layers, with two thin face sheets and a thick and lightweight core in between. The three layers are usually glued to each other. The face sheets have either isotropic in-plane properties (e.g. aluminum foils) or orthotropic properties (e.g. a glass or carbon fibre weave). The core may consist of punctual connections (pole fibres), line connections (corrugations), a full two-dimensional connection (e.g. foam), or a structured line polygonal connection (e.g. a honeycomb core).

  Each of the three constituents has its own uncertain characteristics: geometrical arrangement, precise thickness and material properties. On top of that, the glue layers add a substantial degree of uncertainty too, as it may be difficult to precisely control the amount of glue which is applied at each position. Especially with thin foil honeycomb cores, the area of the wetted surfaces between the cell wall or the face sheet and the adhesive in relation to the volume of adhesive is uncertain, affecting the strength of the bond.

A particular aspect of sandwich materials is the multitude of failure modes which may occur (core shear, face sheet buckling, dimpling, ...). It is unconservative to assume that all constituents remain intact, but it is difficult to identify the position when imperfections occur and to estimate the level of degradation which they bring about.

This overview of diverse processing techniques shows that most processes do not have the capability to guarantee the precise position and orientation of the fibre reinforcement. For some processes, the fibre volume fraction is not constant within the volume of the component either.

Effect of parameter scatter on material stiffness properties The designer has many degrees of freedom, including the selection of raw materials for both the matrix and the fibre reinforcement, the architecture of the fibre reinforcement, the fibre volume fraction, the number of layers and the orientation of layers. For the analyst, this large set of design degrees of freedom translates into a wide range of model parameters, and inevitably also a wide range of uncertain or imprecise material data.

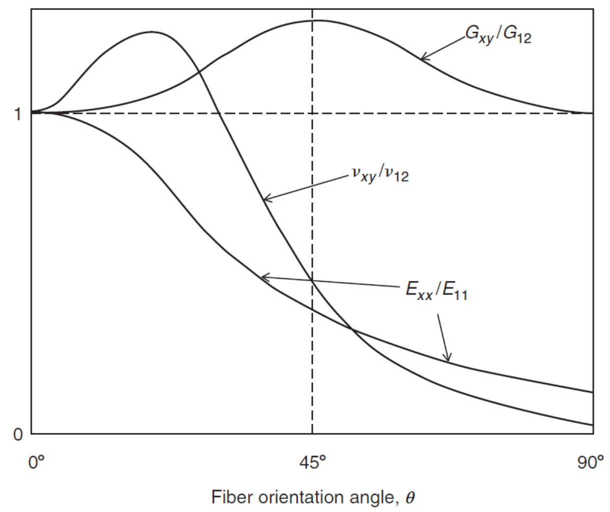
Most composite materials with long fibre architectures exhibit orthotropic behaviour, expressed by Eq.1:

$$\boldsymbol{\epsilon} = \begin{Bmatrix} \epsilon_{11} \\ \epsilon_{22} \\ \epsilon_{33} \\ \epsilon_{12} \\ \epsilon_{23} \\ \epsilon_{31} \end{Bmatrix} = \begin{bmatrix} \frac{1}{E_{11}} & -\frac{\nu_{21}}{E_{22}} & -\frac{\nu_{31}}{E_{33}} & 0 & 0 & 0 \\ -\frac{\nu_{12}}{E_{11}} & \frac{1}{E_{22}} & -\frac{\nu_{32}}{E_{33}} & 0 & 0 & 0 \\ -\frac{\nu_{13}}{E_{11}} & -\frac{\nu_{23}}{E_{22}} & \frac{1}{E_{33}} & 0 & 0 & 0 \\ 0 & 0 & 0 & \frac{1}{2G_{12}} & 0 & 0 \\ 0 & 0 & 0 & 0 & \frac{1}{2G_{23}} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1}{2G_{31}} \end{bmatrix} \begin{Bmatrix} \sigma_{11} \\ \sigma_{22} \\ \sigma_{33} \\ \sigma_{12} \\ \sigma_{23} \\ \sigma_{31} \end{Bmatrix} = \mathbf{D}\boldsymbol{\sigma}. \qquad (1)$$

With ϵ representing strain and σ stress, E_{11}, E_{22} and E_{33} Young's moduli along the direction 1 (aligned with the warp fibre), 2 (aligned with the weft fibre) and 3 (through the thickness). G_{12}, G_{23} and G_{31} represent the shear moduli in the planes 12, 23 and 31. When the load is applied along orientations x and y which include an angle $\theta \neq 0$ with the orientations 1 and 2, the compliance matrix $\mathbf{D}$ in the constitutive relation Eq.1 changes into the matrix $\mathbf{D}'$ as expressed in Eq.2 [6]:

$$\mathbf{D}' = \mathbf{T}\mathbf{D}\mathbf{T}^{-1} \quad \text{with} \quad \mathbf{T} = \begin{bmatrix} \cos^2\theta & \sin^2\theta & 0 & -\sin 2\theta & 0 & 0 \\ \sin^2\theta & \cos^2\theta & 0 & \sin 2\theta & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 \\ -0.5\sin 2\theta & 0.5\sin 2\theta & 0 & \cos 2\theta & 0 & 0 \\ 0 & 0 & 0 & 0 & \cos\theta & \sin\theta \\ 0 & 0 & 0 & 0 & -\sin\theta & \cos\theta \end{bmatrix}. \qquad (2)$$

This relation is used to express the variation of material stiffness constants for a change of orientation of the load. In the application to uncertainty, the misalignment of the fibre orientation with respect to the orientation of loading may be accidental, but the effect is significant, as illustrated by figure 1, which is taken from [7] and [8]. The left hand side of the figure shows the variation of the elastic orthotropic stiffness constants for different orientations of a uniaxially reinforced glass fibre composite lamina with respect to the applied uniaxial tensile load. The graph shows a significant decrease of stiffness with increasing misalignment of the fibre. The right hand side of the figure is valid for a cross-ply (0°-90°) carbon-epoxy system. The graph shows the variation of the Young's modulus for different alignments of the fibre orientations with respect to the loading direction. The graphs show that the equivalent material stiffness depends strongly on the fibre placement. An imprecise placement of the fibre inevitably leads to a change of stiffness with respect to the nominal values. The left hand side of the graph also shows that the orthotropic elastic constants are inter-related.



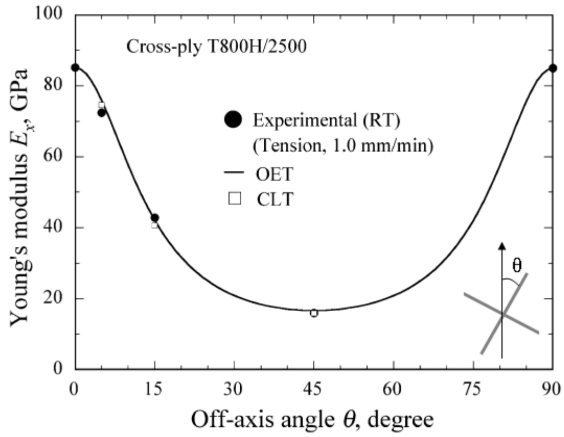


Fig. 1: Dependency of in-plane material parameters on the orientation θ of the major fibre axis to the loading direction; left: variation of the elastic constants of a continuous E-glass fibre lamina [7]; right: variation of the tensile Young's modulus for a cross-ply carbon-epoxy composite [8]

Another geometrical parameter that determines the homogenised stiffness characteristics of a textile composite material is the so-called crimp factor. It is a measure of the waviness of the yarn through the thickness of the panel. A general tendency is that the equivalent modulus of a textile composite increases with decreasing crimp.

Realistic data on material uncertainty and variability

Powerful mathematical tools are available to represent and propagate the effect of uncertainty and variability on finite element models of structural systems. However, there is a severe lack on practical data on real material systems and on concepts to generate reliable and useful data in a format that can be fed into the powerful numerical models that are available. Authors usually content themselves with making assumptions, e.g. on the covariance function of the Young's modulus and the correlation length [2]. They have done sensitivity analyses and they prove that the correlation length has a very large effect on the final result, yet validated data are not available. Charmpis and Schuëller [9] propose two approaches to achieve significant advances in realistic material modelling

1. establishing experimental data on the spatially correlated random fluctuations of uncertain material properties

2. deriving probabilistic information for macroscopic properties from the lower scale mechanical characteristics of materials

Although the need for experimental data was identified already in the 80ies, it appears that no evident step in this direction has been made until a few years ago. The second approach is essentially a multi-scale approach.

Extensive research efforts are currently ongoing to develop a multi-scale modelling procedure at successive scales. Depending on the type of material, the micro-scale describes properties with a reference length in the order of $10^{-6} - 10^{-4}$m, the meso-scale describes properties with a reference length in the order of $10^{-4} - 10^{-2}$m, and entire component structural behaviour is described on the macro-scale, with reference lengths in the order of $10^{-2} - 10^{0}$m and above. The step from a lower level to a higher level is made using homogenisation procedures, that assign overall properties at a higher scale based on lower scale data. So far, these models are mainly deterministic. When these models will be well established, they present an excellent opportunity to introduce variability at the appropriate level, and to predict the propagation of their effect to a higher level, and ultimately to the entire component. Experiments will however always be required to validate these models.

Multi-scale models also have the advantage that spatial variation of homogenised properties can be described based on lower scale characteristics. This presents opportunities for realistic quantification of random fields, for which experimental data are currently missing.

Quantification of variability on composite geometry in carbon fibre weaves

The objective of this section is to develop a consistent modelling strategy which can be used to generate virtual samples of a composite reinforcement architecture. The statistics of the set of samples which are generated should match the statistics of a typical real composite panel. Figure 2 summarises

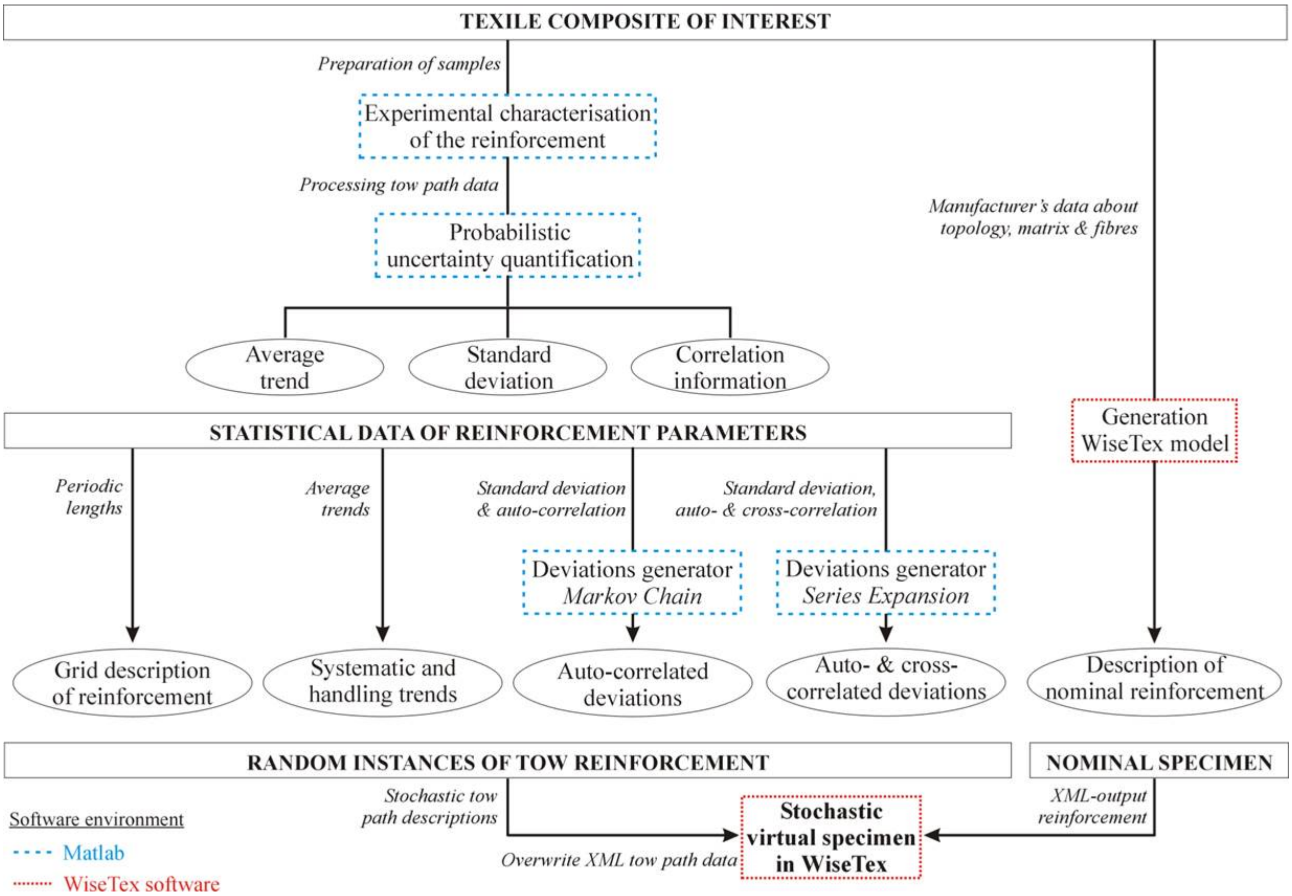


Fig. 2: The general methodology for modelling data uncertainty in composite reinforcement geometry consists of three steps: 1 (top)- acquisition of geometry data; 2 (middle)- processing of statistical data and correlation within one tow and between adjacent tows; 3 (bottom)- generation of virtual tow realisations which have the same statistics as the experimental data set

the entire procedure.

Modelling strategy Realistic woven specimens are acquired that are replicas of experimental samples. Randomness is introduced in the numerical models at the meso- and macro-level; scatter in the matrix and fibre properties are not considered. Variability of each tow path is defined for the centroid co-ordinates (x, y, z), tow aspect ratio AR, tow area A and orientation θ in cross-section which fully describe a woven reinforcement. Figure 2 presents an overview of the multi-scale framework, where three main steps can be distinguished to obtain such random representations:

1. The first step is the collection of experimental data and the subsequent statistical analysis, according to the procedure proposed by Bale et al. [10]. Two length scales are considered, one on the level of the representative volume element using μCT and the other on the scale of multiple unit cells. The former set gives precise positional and cross-sectional data on the meso-level, identifying full details of 3D geometry. The latter data set is required to describe long-scale deviations from the nominal reinforcement pattern.

   (a) Characterisation of the short-range scatter (meso-scale) with samples close to the unit cell size.

   (b) Characterisation of the long-range variation (macro-scale) with samples spanning several unit cells.

   (c) Statistical analysis of the tow path parameters in terms of average trends, standard deviation and correlation lengths. For each parameter, the average trend $\langle \epsilon_i^{j,t,p} \rangle$ and the local deviation $\delta\epsilon_i^{j,t,p}$ are separated:

   $$\epsilon_i^{j,t,p} = \langle \epsilon_i^{j,t,p} \rangle + \delta\epsilon_i^{j,t,p}. \tag{3}$$

   where i refers to the location, j to the tow number, t to the tow genus (warp or weft) and p to the ply or sample.

   Vanaerschot et al. [11] give full details on the analysis procedure and statistical data processing.

2. The second step is the stochastic multi-scale modelling of the reinforcement architecture that is experimentally identified in step 1.

   (a) Definition of systematic and handling trends from the experimental data.

   (b) Generation of zero-mean deviations correlated along the tow path using the Monte Carlo Markov chain method.

   (c) Generation of zero-mean deviations correlated along and between neighbouring tow paths using the cross-correlated series expansion technique.

   For the generation of geometry data, two options are available. When cross-correlation between adjacent tows are not taken into account, the procedure by Blacklock et al. [12] is used. The representation is done using a Markov process. Each tow parameter is generated in an independent way, and for the calibration step, standard deviation and the nearest neighbour correlation information is used. When cross-correlations are taken into account, a cross-correlated series expansion is used, based on Karhunen-Loève decomposition, as proposed by Vorechovský et al. [13, 14]. Vanaerschot et al. [15] give full details on the generation of an approximated random field.

3. Construction of virtual specimens in the WiseTex software

   (a) Simulation of the nominal model with matrix and fibre properties from the manufacturer.

   (b) Redefinition of the reinforcement information with the produced tow paths.

   (c) Recalculations of the path orientation vectors and length, in addition to the updated general unit cell properties.

   The WiseTex [17] software suite is developed at the Materials Engineering Department of KU Leuven. It is a pre-processor for the generation of virtual 3D models of a wide range of textile composite architectures. The pre-processor prepares for the generation of finite element models for the mechanical analysis of composite structures.

This step of the analysis generates virtual specimens which have the same statistical characteristics as the samples on which the experimental data acquisition was done. For each virtual sample, a WiseTex model is generated, with the original nominal tow path being overwritten by updated paths. Discrete random tow path realisations are interpolated and accompanied with information on the orientation vectors of each tow cross-section, the path length for each segment between two discrete locations and with new, off-nominal unit cell dimensions and properties.

Vanaerschot et al. [16] present details of the procedure for generating virtual samples.

The entire methodology allows for the generation of a set of statistically relevant samples. In a subsequent analysis step, these models are used for the investigation of the homogenised properties of a composite panel.

When other topologies than woven are considered, additional parameters should be quantified to allow a full description, e.g. the braid angle for braids and the distortion of the z-yarn in case of non-crimp fabrics.

Application to a carbon fibre twill weave

This section presents an application to the methodology which is described above. For a first validation experiment, a composite architecture is selected which is expected to exhibit only a low to moderate degree of geometrical variability.

Sample data The entire procedure is applied on a polymer textile composite with a twill 2/2 woven topology (Hexcel®G0986 Injectex), with 6K carbon fibre AS4C tows. The matrix is an expoxy resin Epikote®828LVEL with Dytek®DCH-99 hardener. The samples are prepared in a Resin Transfer Moulding (RTM) process and the unit cell size is 11.4mm×11.4mm. Some of the samples on which data have been acquired have 7 layers, others have only a single layer.

Geometry data acquisition Two sets of measurements have been conducted on two different types of samples of the composite. For the characterisation of short-range scatter μCT scans have been taken on samples with dimensions close to the unit cell size. These samples have 7 layers. Olave et al. [18] discuss the details of this measurement campaign. For the characterisation of longe-range scatter optical scans have been taken on samples with dimensions equal to several unit cell size. These samples have only one layer.

Statistical processing of tow data Statistical analysis is conducted on the date of each individual tow. The average trend in the figure exhibits a wavy pattern, marking the crossing of one warp tow over or under a weft tow.

Modelling of tow data In the modelling step, models are generated which extend over 10 unit cells, with the length of one unit cell being represented by 32 equi-distant points. Short-range periodic trends and long-range handling trends are combined. Figure 3 shows the average trends for the warp and weft genus, as they are measured in the experimental μCT analysis, for each of the five tow attributes. It was found that simulated trends correspond to experimental trends when cross-correlation is taken into account. Correlation is verified comparing a set of generated tow paths to a set of measured paths. For the out-of-plane and for the in-plane transverse centroid co-ordinates visual comparison is easy. Another element of comparison is a histogramme. Both methods of comparison show a very good match between simulations and experiments.

Generation of virtual models In the final step, 3D models are generated in the WiseTex preprocessor. 3D models allow for straightforward visual comparison, and models are found to actually exhibit the appropriate degree of spatial scatter.

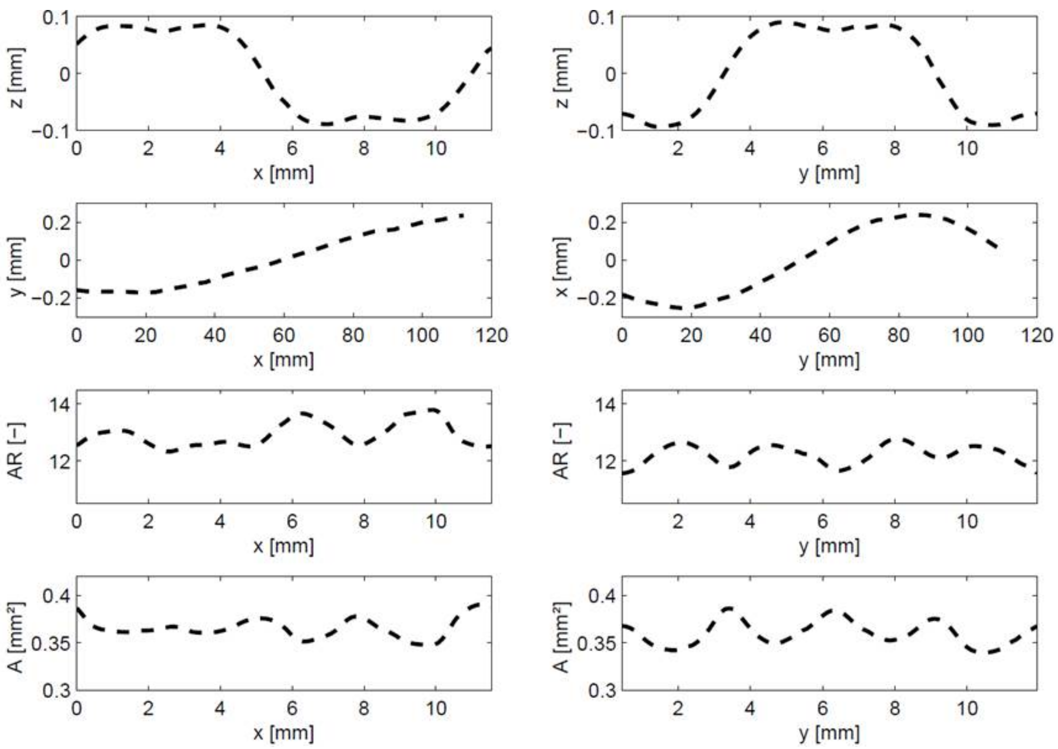


Fig. 3: Trends on each of the tow attributes as they result from the μCT analysis: the left hand graphs apply for the warp genus, and the right hand column applies to the weft genus; the top line graphs show the out-of-plane position of the tow centroid for one unit cell; the second line graphs represent the lateral in-plane deviation of the tow centroid for 10 unit cells; the third and fourth line graphs show the aspect ratio and the cross-section, respectively, each for one unit cell

Conclusions

This research is an effort to characterise uncertainty and variability in composite materials. The methodology is demonstrated for a carbon-epoxy 2/2 twill woven composite. The comparison between experimental and simulated deviation trends is found to be good. Randomly generated virtual specimens possess the target statistical information.

With the multitude of composite fibre architectures and processing methods, a lot of research work is still required to validate the proposed procedure also for other composite materials. A flat 2/2 twill woven composite has in theory a highly regular reinforcement pattern, but a curved geometry or braided or knitted architecture add up substantially to the complexity of the process and probably also to the degree of variability in the product. Another aspect is the size of the product, with parts in the order of several metres having much more variability than the unit cell size and the small components which have been investigated in this work.

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